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SPECIFICATION FOR RHODIUM SALTS FOR ELECTROPLATING

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Indian Standard

SPECIFICATION FOR RHODIUM SALTS FOR ELECTROPLATING

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Indian Standard

SPECIFICATION FOR RHODIUM SALTS FOR ELECTROPLATING

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 10 August 1981, after the draft finalized by the Electroplating Chemicals Sectional Committee had been approved by the Chemical Division Council.

0.2 General applications of rhodium plating in the industrial field are well known. Essential properties of the electro-deposited rhodium metal are high hardness (hence good wear-resistance) and resistance to corrosion and tarnishing along with good reflectivity. Rhodium electrodeposits have been used in the jewellery trade for many years for protecting silver from tarnishing. Rhodium is also used in the in-printed electronic circuitry, where wiping contacts are provided. In recent times, the main use of rhodium has been for electrical, sliding contacts and contacts required to operate after periods of idleness. In the fields of electronics and telecommunications, where electrical signals are small and voltages insufficient to break down tarnish films on silver or base metal contacts, rhodium deposit is used. Rhodium is also used for short-time protection in high temperature work. The thickness of rhodium plating in such cases is generally of the order of 2·5 to 5 micrometers (μm). Thicker plating of rhodium (about 50 μm) is used on iridium crucibles. However, deposits thicker than 12·5 μm are rarely used, since rhodium is one of the lighter metals in the platinum group (RD 12·4).

0.3 A number of plating baths are used for rhodium plating. The commercial baths are fairly strong acid solutions of rhodium phosphate or rhodium sulphate. Sulphamic and fluoboric acids are also used for preparing rhodium plating baths, but they are more of academic interest. For deposits for protection of jewellery, rhodium phosphate solution is used, while for industrial use, rhodium sulphate solution is preferred. Sometimes phosphate-sulphate baths are also used for depositing rhodium. Rhodium can be directly plated on brass, nickel, nickel-silver, phosphor-bronze, beryllium, copper, etc. Because of the acid content of the bath and the position of rhodium in the electrochemical series, it cannot be plated directly on to iron, steel, zinc, aluminium, lead or tin and, therefore, a preliminary deposit of silver is usually needed on these metals. The electro-deposited rhodium has a hardness of about 800 VPN.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2 - 1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for rhodium salts (sulphate and phosphate) for electroplating.

2. REQUIREMENTS

2.1 Rhodium sulphate shall be in the form of a concentrated solution, in dilute sulphuric acid, and free from foreign matter and visible impurities.

2.2 Rhodium phosphate shall be in the form of a concentrated solution, brownish in colour and free from foreign matter and visible impurities.

2.3 The solutions of rhodium salts for electroplating shall also comply with the requirements given in Table 1, when tested in accordance with the methods prescribed in Appendix A. Reference to the relevant clauses of Appendix A is given in col 5 of Table 1.

3. PACKING AND MARKING

3.1 Packing — Rhodium salt solutions for electroplating shall be packed in high density polyethylene bottles of 25, 50 or 100 ml capacities (to contain solutions equivalent to 2 g, 4 g and 8 g respectively of rhodium) or as agreed to between the purchaser and the supplier.

3.2 Marking — The bottles shall be marked with the following:

a) Name of the material and mass-equivalent to rhodium (as Rh) in the container;

b) Name of the manufacturer or his recognized trade-mark, if any; and

c) Date of manufacture and batch number to enable the lot to be traced from records.

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*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR RHODIUM SALTS FOR ELECTROPLATING

(*Clause* 2.3)

SL No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST (REF TO CL NO. IN APPENDIX A)
		Rhodium Sulphate Solution	Rhodium Phosphate Solution	
(1)	(2)	(3)	(4)	(5)
i)	*p*H of the solution as received, not less than	2	1·5	A-2
ii)	Rhodium (as Rh), g/l, *Min*	80	80	A-3
iii)	Sulphate (as SO_4), g/l, *Min*	120	—	A-4
iv)	Phosphate (as PO_4), g/l, *Min*	—	80	A-5
v)	Silver (as Ag), g/l, *Max*	0·03	0·02	A-6
vi)	Lead (as Pb), g/l, *Max*	0·03	0·02	A-7
vii)	Copper (as Cu), g/l, *Max*	0·03	0·02	A-8
viii)	Iron (as Fe), g/l, *Max*	0·03	0·02	A-9

3.2.1 The bottles may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for its conformity with the requirements of this standard shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.3)

METHODS OF TEST FOR RHODIUM SALTS FOR ELECTROPLATING

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemical and distilled water (*see* IS : 1070-1977*) shall be used in the tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF *p*H

A-2.1 Procedure — Determine the *p*H of the concentrated solution, as received, at a temperature of 27 ± 2°C with the help of a *p*H meter, using glass electrodes.

A-3. DETERMINATION OF RHODIUM

A-3.1 General — The sample solution is made alkaline with potassium hydroxide and precipitation carried out using hydrazine sulphate whereafter the precipitate is ignited/dried to constant mass after adding a few drops of formic acid and weighed as rhodium metal.

A-3.1 Reagents

A-3.1.1 *Dilute Sulphuric Acid* — 6 percent (*v/v*).

A-3.1.2 *Sodium Hydroxide Solution* — 20 percent (*m/v*).

A-3.1.3 *Hydrazine Sulphate Solution* — saturated aqueous solution.

A-3.1.4 *Dilute Nitric Acid* — 10 percent (*v/v*).

A-3.2 Procedure — Pipette 2·0 ml of the sample solution into a 250 ml-beaker and dilute to about 100 ml with water. Add 50 ml of sodium hydroxide solution followed by 30 ml of hydrazine sulphate solution with stirring. Heat gently on a hot plate. When evolution of gas bubbles ceases add another 20 ml of hot hydrazine sulphate solution and allow to digest on the hot plate for 1 hour. Then filter the precipitate through a filter paper (Whatman No. 42 is suitable) and wash thoroughly with water. Transfer the precipitate together with the filter paper to the

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*Specification for water for general laboratory use (*second revision*).

original beaker, add some dilute nitric acid and warm gently. Filter again, wash thoroughly with hot water and ignite in a crucible. Add three drops of formic acid and dry to a constant mass at 110 ± 2°C. Cool in a desiccator and weigh. Preserve the filtrate for further tests.

A-3.3 Calculation

$$\text{Rhodium (as Rh), g/l} = M \times 500$$

where

M = mass in g of the ignited and dried precipitate.

A-4. DETERMINATION OF SULPHATE

A-4.1 Reagents

A-4.1.1 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976*.

A-4.1.2 *Barium Chloride Solution* — 20 percent (*m/v*) in water.

A-4.1.3 *Dilute Hydrochloric Acid* — 1 percent (*v/v*).

A-4.2 Procedure — Pipette 2 ml of the sample solution in a 100-ml beaker, add 10 ml of concentrated hydrochloric acid, boil for 5 min, dilute to 50 ml with water and again heat almost to boiling. Take 80 ml of barium chloride solution and 2 ml of concentrated hydrochloric acid in a 250-ml beaker, heat almost to boiling and pour the contents of the 100-ml beaker into it with stirring. Add a drop of barium chloride solution to be certain that the precipitation is complete. Rinse out the 100-ml beaker into the 250-ml beaker with five 10-ml portions of hot water. Leave on a hot plate for 1 h and filter through a hard filter paper (Whatman No. 42 is suitable), washing the precipitate with dilute hydrochloric acid and then with water. Transfer the filter paper to a tared crucible, dry and ignite, gradually raising the temperature and maintaining it at 600°C for at least 1 hour.

A-4.3 Calculation

$$\text{Sulphate (as } SO_4 \text{), g/l} = 205{\cdot}8\, M$$

where

M = mass in g of the ignited precipitate.

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*Specification for hydrochloric acid (*second revision*).

A-5. DETERMINATION OF PHOSPHATE

A-5.1 Reagents

A-5.1.1 *Concentrated Nitric Acid* — *See* IS : 264-1976*.

A-5.1.2 *Concentrated Sulphuric Acid* — *See* IS : 266-1977†.

A-5.1.3 *Phenolphthalein Indicator Solution* — Prepare by dissolving 0·1 g of phenolphthalein in 100 ml of 60 percent (*v/v*) rectified spirit.

A-5.1.4 *Ammonium Hydroxide* — RD 0·9.

A-5.1.5 *Concentrated Hydrochloric Acid* — *See* IS : 265-1976‡.

A-5.1.6 *Ammonium Acetate* — Solid.

A-5.1.7 *Magnesia Mixture* — Dissolve 55 g of magnesium chloride ($MgCl_2.6H_2O$) and 105 g of ammonium chloride in about 800 ml of water and then dilute to 1 000 ml with water.

A-5.1.8 *Dilute Ammonia* — 1 : 2 (*v/v*).

A-5.1.9 *Ammonium Nitrate Solution* — Saturated solution in water.

A-5.2 Procedure — To the filtrate preserved in **A-3.2**, add 5 ml each of concentrated nitric and sulphuric acid. Boil the solution and reduce the volume to about 40 ml. Cool and dilute with water to about 100 ml. Add 5 to 10 drops of phenolphthalein indicator solution followed by ammonium hydroxide slowly with stirring until the colour of the solution changes to pink. Slowly add concentrated hydrochloric acid drop by drop until the pink colour disappears and then add 10 ml of the acid in excess. Add 5 g of ammonium acetate and stir in 50 ml of the magnesia mixture. Heat to boiling and slowly add ammonium hydroxide until the solution turns pink again. Stir the solution for about 5 min until the precipitate which first forms becomes finely crystalline in structure. Cool to room temperature and add ammonium hydroxide equivalent to one-fifth of the volume of the solution. Allow to stand for at least 4 h or overnight. Filter through a hard filter paper (Whatman No. 44 is suitable), washing the precipitate with 10 ml portions of cold dilute ammonia till free from chloride. Moisten the filter paper with ammonium nitrate solution, dry the precipitate in a tared platinum crucible. Char the paper slowly, burn off carbon at as low a temperature as possible with free access of air. Finally ignite to 1 000 to 1 100°C over a meker burner for 20 to 30 min. Cool in a desiccator and weigh to a constant mass.

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*Specification for nitric acid (*second revision*).

†Specification for sulphuric acid (*second revision*).

‡Specification for hydrochloric acid (*second revision*).

A-5.3 Calculation

$$\text{Phosphate (as } PO_4 \text{), g/l} = 426{\cdot}75\ M$$

where

M = mass in g of the ignited precipitate.

A-6. DETERMINATION OF SILVER

A-6.0 General — Silver gives a red-violet precipitate with *p*-dimethyl-amino-benzylidene-rhodanine in an acidic medium. Interference by metal ions like mercury, copper, gold, palladium and lead may be prevented by treating the precipitate with 5 percent potassium cyanide solution and filtering.

A-6.1 Reagents

A-6.1.1 *p-Dimethylamino-Benzylidene-Rhodanine Solution*

a) *For precipitation* — Dissolve 20 mg of the reagent in 100 ml of ethyl alcohol.

b) *For standards* — Dissolve 20 mg of the reagent, crystallized from alcohol, in 100 ml of 0·01 *N* sodium hydroxide solution.

A-6.1.2 *Potassium Cyanide Solution* — Dissolve 5 g of potassium cyanide in 100 ml of 0·001 *N* sodium hydroxide solution.

A-6.1.3 *Ethyl Alcohol*

A-6.2 Procedure — Pipette 1·0 ml or a smaller quantity of solution, depending upon requirement, of the sample solution into a 100-ml beaker, dilute with water to about 5 ml, adjust the concentration of sulphuric acid to 0·4 *N* and add 5 ml of alcoholic rhodanine solution (For less than 10 mg of silver, make the precipitation in 0·1 *N* acid concentration and use smaller volumes, so that the final solution can be diluted to 25 ml). Allow the solution to stand in the dark for at least 2 h (preferably overnight). Filter through a sintered glass crucible (G No. 4) and wash with water. Place the crucible in the beaker in which precipitation was done and pour 40 ml of ethyl alcohol into the crucible and beaker. Cover with a watch-glass and boil very gently for 15 minutes. Suck off the alcohol in the crucible and wash the crucible and the beaker with cold alcohol, till the last portion is entirely colourless. Empty and clean the suction flask.

A-6.2.1 Pour 5 ml of potassium cyanide solution into the precipitation beaker and then into the crucible, using a stirring rod to distribute the solution over the walls of the vessels. Draw off the solution and wash with a little water. Repeat the treatment with 5 ml of cyanide solution. Transfer the solution from the suction flask to a 50 ml volumetric flask and make up the volume. Without delay obtain the transmittance of the solution with the aid of a blue filter (460 nm).

A-6.2.2 Prepare the standard curve by measuring out 0·1 ml, 0·2 ml, 0·3 ml and 1 ml of the aqueous rhodanine solution, diluting to 50 ml with 0·000 1 *N* sodium hydroxide solution, and finding the transmittance. One part of rhodanine corresponds to 0·408 part of silver.

A-7. DETERMINATION OF LEAD

A-7.1 Reagents

A-7.1.1 *Sodium Acetate* — solid.

A-7.1.2 *Glacial Acetic Acid*

A-7.1.3 *Potassium Chromate Solution* — 1 percent (*m/v*) in water.

A-7.1.4 *Standard Lead Solution* — Dissolve 0·160 g of lead nitrate in 100 ml of water containing 1 ml of concentrated nitric acid (*see* Note) and dilute with waterup to the mark in a 1 000-ml volumetric flask. Dilute 10 ml of this solution with water up to the mark in a 100-ml volumetric flask. One millilitre of this diluted solution is equivalent to 0·01 mg of lead (as Pb).

NOTE — Add silver nitrate equivalent to the quantity of silver obtained in **A-6** to compensate for any interference by silver.

A-7.2 Procedure — Pipette 1·0 ml of the sample solution into a 100-ml Nessler cylinder, dilute with water to 10 ml and dissolve 1 g of sodium acetate. Add five drops of glacial acetic acid followed by 1 ml of potassium chromate solution, dilute with water up to the mark and mix. Carry out a control test in another Nessler cylinder using the same quantities of reagents as with the sample and the following volumes of standard lead solution in place of the material.

Name of the Material	*Volume of Standard Lead Solution* ml
Rhodium sulphate solution	3
Rhodium phosphate solution	2

A-7.2.1 The requirement of the test shall be taken as having been satisfied if the turbidity produced with the material is not deeper than that produced in the control test.

A-8. DETERMINATION OF COPPER

A-8.0 General — α-Benzoinoxime (cupron) yields a green precipitate with copper in dilute ammoniacal solution. Ions which are precipitated by ammonium hydroxide are kept in solution by the addition of ammonium tartrate to make the reagent specific for copper.

A-8.1 Reagents

A-8.1.1 *α-Benzoinoxime Solution* — 2 percent (*m / v*) solution in rectified spirit.

A-8.1.2 *Dilute Ammonium Hydroxide Solution* — 1 : 4 (*v/v*).

A-8.1.3 *Absolute Alcohol.*

A-8.2 Procedure — Pipette 1·0 ml of the sample solution into a 100-ml beaker and dilute to 25 ml with water. Neutralize with ammonia, acidify with tartaric acid and add 10 ml of α-benzoinoxime solution. Heat to boiling and filter the precipitate through a tared sintered glass crucible (G No. 4). Wash with hot 1 percent (*v/v*) ammonium hydroxide solution, then with alcohol and finally with hot water. Dry to constant mass at 105 to 110°C.

A-8.3 Calculation

$$\text{Copper (as Cu), g/l} = 220{\cdot}1\, M$$

where

M = mass in g of the precipitate.

A-9. DETERMINATION OF IRON

A-9.0 General — Aqueous solution of 7-iodo-8-hydroxyquinoline-5-sulphonic acid gives a stable green colour with ferric ions; the intensity of the colour varies with the concentration of ferric ions.

NOTE — The reaction is not given by ferrous ions, although it has been stated that under certain conditions a colour is produced as a result of auto-oxidation to the ferric state. When one part of ferric ion in 5 million parts of solution is present, the green colour is just distinguishable from the yellow tint of the reagent.

A-9.1 Apparatus

A-9.1.1 *Nessler Cylinders* — Two of 50-ml capacity.

A-9.2 Reagents

A-9.2.1 *Buffer Solution* — Prepare buffer of *p*H 2·5 by mixing 25 ml of 0·2 *M* potassium hydrogen phthalate (40·83 g per litre solution) with

36·3 ml of 0·1 *N* hydrochloric acid (10 ml of concentrated hydrochloric acid diluted with 1 litre water) and making them up to 100 ml in a measuring flask.

A-9.2.2 *7-Iodo-8-Hydroxyquinoline-5-Sulphonic Acid Solution* — Dissolve 0·2 g of the reagent in 100 ml of water.

A-9.2.3 *Ammonium Persulphate*

A-9.2.4 *Standard Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate [$FeSO_4(NH_4)_2SO_4 6H_2O$] in about 100 ml of water containing 10 ml of dilute sulphuric acid (10 percent (*v/v*) and dilute to one litre. Take 10 ml of this solution and dilute to 100 ml. One millilitre of this diluted solution contains 0·01 mg of iron (as Fe).

A-9.3 Procedure — Pipette 1·0 ml of the sample solution and 25 ml of water into a 50-ml Nessler cylinder and mix. Add 10 ml of the buffer solution and a pinch of ammonium persulphate followed by five drops of 7-iodo-8-hydroxyquinoline-5-sulphonic acid solution and make the solution up to the mark. Carry out a control test in another Nessler cylinder using the same quantity of reagents as with the material and the following volumes of standard iron solution in place of the material:

Name of the Material	*Volume of Standard Iron Solution* ml
Rhodium sulphate solution	3
Rhodium phosphate solution	2

NOTE — The test should be carried out in an acidic medium having *p*H about 2·5. The test may be applied in the presence of all metals having colourless ions, with the exception of cupric salts. Coloured ions do not interfere unless present in sufficient amount to mar the green colour produced by the reaction.

A-9.3.1 The material shall be taken as having satisfied the requirement of the test if the intensity of colour produced with the material is not greater than that produced in the control test.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF RHODIUM SALTS FOR ELECTROPLATING

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

B-1.1 Samples shall be taken in a protected place not exposed to damp air, dust or soot.

B-1.2 The sampling instruments shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in suitable, clean, dry and air-tight containers on which the material has no action.

B-1.6 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.7 Each sample container shall be sealed air-tight after filling, and marked with full details of sampling and the date of sampling and details given in **3.2**.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material of the same type drawn from a single batch of manufacture shall constitute the lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

B-2.2 For ascertaining the conformity of the material in any lot to the requirements of this specification, samples shall be tested for each lot separately.

B-2.3 The number of containers to be selected at random from a lot shall depend upon the lot size and shall be in accordance with col 1 and 2 of Table 2.

B-2.3.1 In order to ensure the randomness of selection, procedures given in IS : 4905-1968* may be followed.

B-3. INDIVIDUAL SAMPLES AND COMPOSITE SAMPLES

B-3.1 From each of the containers selected as in **B-2.3,** a representative portion of the material shall be drawn using a suitable sampling implement. The total quantity so drawn from each of the containers shall be approximately equal to thrice the quantity required for testing purposes.

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*Methods for random sampling.

TABLE 2 SCALE OF SAMPLING

(*Clause* B-2.3)

LOT SIZE (N) (1)	NUMBER OF CONTAINERS TO BE SELECTED (n) (2)
Up to 25	2
26 ,, 50	3
51 ,, 100	4
101 and above	5

B-3.2 From the samples (**B-3.1**) representing different containers, a small but approximately equal quantity of the material shall be taken and mixed thoroughly to form a composite sample sufficient to carry out testing of the characteristics specified. The composite samples so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

B-3.3 The remaining portions of the material in sample (**B-3.1**) from different containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the n containers selected shall be for the purchaser, another for the supplier and the third for the referee.

B-3.4 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight and labelled with full identification particulars given in **B-1.7**.

B-3.5 A referee sample consisting of a composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

B-4. NUMBER OF TESTS CRITERIA FOR CONFORMITY

B-4.1 Test for the determination of rhodium (as Rh) shall be carried out on any two of the individual samples.

B-4.2 Tests for the remaining characteristics shall be done on the composite sample.

B-4.3 The lot shall be deemed to conform to this specification if the test results on individual and composite samples satisfy the corresponding requirements given in Table 1.